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This article was submitted to
22nd Aging, Compatibility and Stockpile Stewardship Conference,
Oak Ridge, TN, April 27-30, 1999

August 24, 1999

U.S. Department of Energy

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Summary

A preliminary compatibility study was carried out between the plastic-bonded PETN-based high explosive LX-16 and the adhesive Halothane 73-18. The work, based on the Chemical Reactivity Test (CRT), used non-standard times and temperatures to find conditions corresponding to accelerated decomposition. This study is a prequel to a more comprehensive isothermal and thermal cycling study that will include both material evaluation and test fire.

Introduction

The Chemical Reactivity Test (CRT) is the standard test used by LLNL to determine compatibility of high explosive (HE) materials.^{(1),(2)} Reactivity tests are used to determine the reactivity of materials which are in contact with HEs in a weapon or during the fabrication of a weapon component. This test is performed by chromatographically analyzing and measuring the gases liberated from 50:50 mixtures of the high explosive with the material of interest. Each material is tested separately as controls. A measure of the reactivity is obtained when the types and volumes of gases liberated from the mixture are compared with that evolved by the controls.

Chemical reactivity tests under standard CRT conditions (120°C for 22 hrs) at EG&G Mound⁽³⁾ had previously indicated a potential incompatibility between LX-16, a PETN-based plastic-bonded explosive (PBX) used in detonators, and an adhesive (Halothane 73-18). The LX-16 formulation consists of 96.5% by wt. nitroester explosive (PETN)¹ and 3.5 ± 0.5 % by wt. FPC-461 binder. The binder (from Firestone Plastic Company) is a co-polymer of vinyl chloride and chlorofluoroethylene. However, as is typical for PETN compatibility testing, when the temperature was reduced (100°C, 80°C), there was a substantial decrease in total gas volume evolved. The mix of LX-16 and Halothane 73-18 is considered suitable for weapon applications.

Limited aging and compatibility data exist for LX-16. Typically short-term, high temperature compatibility studies or short-term aging studies have been carried out on PBXs to verify stability and/or compatibility. One definitive study⁽⁴⁾ did evaluate basic LX-16 compatibility issues with other detonator materials. Isothermal heating of materials in these mini "Core" sample containers was conducted at 60, 80, and 100°C for periods of 36, 71 and 102 days. Gas samples were collected at 30, 60 and 90 days and analyzed by gas chromatography (GC).

This study did not reveal unexpected material interactions or changes in behavior under the conditions of the study. Some of the materials, however, were not the final choice for weapon use (MSAD, or Mechanical Safe and Arm Device). Most notable of the differences was lack of adhesive between the LX-16 and the aluminum can. The final choice of adhesive was Halothane 73-18.

¹ PETN = Pentaerythritol Tetranitrate

This type of thermal aging study is intended as a screening test to identify immediate compatibility issues and aid in the selection of materials for final design. It is not meant to be a definitive probe for long-term aging behavior, nor a method to quantitatively predict the useful life of a material. In a complex material like a PBX, some thermal-driven reaction pathways may dominate at high temperatures that may otherwise proceed at a negligible rate at lower temperatures.

To gain further insight into the thermal degradation behavior of LX-16 and adjacent detonator materials, a preliminary compatibility study was carried out at Mason & Hanger, Pantex Plant. The temperature and duration of heating was varied to find conditions under which the LX-16 and the adhesive Halthane 73-18 showed a significant increase in gas evolution. These conditions were then used to provide test matrix limits for a second, more complete aging and compatibility study to be carried out at Pantex.

Experimental

An evaluation of the compatibility of LX-16 and Halthane 73-18 was carried out on the Pantex Plant CRT apparatus.⁽⁶⁾ The tests were carried out with 250-mg samples, individually or per component in the 50:50 mixture. Air was removed from the sample holders by purging with helium gas. The sample holders were held within ± 1 °C of the target temperature (60, 80, 100, or 120°C), heating for duration of 22, 45, 70 or 94 hours.

After each sample was cooled down to room temperature, the gases evolved from the control were flushed out and analyzed chromatographically. A gas chromatograph was used to analyze the gas samples. The peaks observed on the chromatograms (O_2 , N_2 , NO, CO_2 , N_2O and CO_2) were identified on the basis of the observed retention times and sequence of appearance. The volumes (in cc) corresponding to the observed peak areas were determined with the aid of previously established calibration curves.

Although tests were carried out on three lots of LX-16 most relevant to weapon MSAD development and manufacture (2194-200-001, 3004-200-001, 89053-200-001), the focus of this study was on the third, the most recently manufactured. The time/temperature matrix and total gas evolved is shown in Table I.

Additional evaluation was carried out on at 80°C and 100°C with LX-16 pellet can assemblies (i.e., aluminum can with LX-16 pellet bonded by Halthane 73-18). Although this test did not rely on the standard CRT 50:50 mix of the two components, it did provide the appropriate material interfaces directly relevant to detonator conditions. When the pellets were removed from their aluminum cans after the test, it was noted that the LX-16 pellet surface had swelled. Both the LX-16 pellet and the Halthane had discolored brown and red-brown respectively. The pellet control sample when removed from its can was flawless white, and the Halthane clear as if new.

Discussion

There appears to be a high temperature reaction between PETN and the Halthane adhesive (a “PUR”, or polyurethane). The greatest degree of gas evolution occurred for temperature $\geq 100^\circ\text{C}$. The greatest increase in gas volumes from 100 to 120°C were seen for carbon dioxide, nitrogen, and nitrous oxide (NO_2) (see Table II).

According to researchers at LLNL,^(7a,b) the experience with PETN and its various formulations in CRT is that it often generates excessive gas in the “standard” 120°C test with other test materials. However, the LLNL material specification for CRT evaluation (RM251808) does allow for lower test temperature of 100°C to be used for compatibility evaluation of PETN formulations. The standard PETN formulations (LX-16, LX-13) usually give acceptable results at this temperature.

Another review of CRT records at LLNL and Pantex for three types of PURs and four types of HE revealed that greater interaction had occurred between the PURs and nitroesters.^(7b) Thermal decomposition of a nitroester like PETN occurs initially through scissioning of the R- ONO_2 bonds. If the PETN decomposes, as suggested by appearance and the CRT data, then the Halthane could undergo oxidation by initially evolved NO_2 .

A second mechanism has also been proposed.^(7b) Since NO₂ oxidizes organic residues with formation of water, the presence of moisture with NO₂ could form nitric acid. It was proposed that the decomposition rates could increase with nitric acid concentration, accelerating the degradation process. Unfortunately, the CRT measurements reported here did not provide an accurate measure of moisture generated.

It is hoped that the proposed hypotheses will be more adequately exercised with the upcoming LX-16 and Halothane thermal aging study at Pantex. This study will be using both isothermal and thermal-cycling conditions to drive degradation for up to a year. Both characterization and test fire will be used to evaluate the effects of degradation.

Table I - Test Matrix and Average Total Evolved Gas (in cc)

	Temperature			
	120°C	100°C	80°C	60°C
22 hrs				
LX-16	0.290	0.014	0.034	
Halothane 73-18	0.042	0.023	0.016	
LX-16/Halothane 73-18	2.517	0.043	0.018	
46 hrs				
LX-16		0.028	0.013	0.013
Halothane 73-18		0.044	0.019	0.018
LX-16/Halothane 73-18		0.206	0.025	0.018
70 hrs				
LX-16		0.022	0.021	0.018
Halothane 73-18		0.028	0.035	0.022
LX-16/Halothane 73-18		0.155	0.026	0.019
94 hrs				
LX-16			0.022	0.023
Halothane 73-18			0.035	0.035
LX-16/Halothane 73-18			0.035	0.034

Table II - Evolved Gases (in cc) for 50:50 Mixtures of LX-16 and Halothane 73-18

22 hours								
temp (°C)	temp (K)	1/T	total gas	N2	CO	NO	CO2	N2O
120	393	2.54	2.517	0.599	0.132	0.166	1.054	0.475
100	373	2.68	0.043	0.024	0.000	0.005	0.014	0.000
80	353	2.83	0.018	0.013	0.000	0.000	0.005	0.000
60	333	3.00						
46 hours								
temp (°C)	temp (K)	1/T	total gas	N2	CO	NO	CO2	N2O
120	393	2.54						
100	373	2.68	0.206	0.081	0.004	0.015	0.092	0.009
80	353	2.83	0.025	0.016	0.000	0.000	0.008	0.000
60	333	3.00	0.018	0.011	0.000	0.000	0.004	0.000
70 hours								
temp (°C)	temp (K)	1/T	total gas	N2	CO	NO	CO2	N2O
120	393	2.54						
100	373	2.68	0.155	0.075	0.000	0.008	0.068	0.007
80	353	2.83	0.026	0.016	0.000	0.000	0.008	0.000
60	333	3.00	0.019	0.010	0.000	0.000	0.004	0.000
94 hours								
temp (°C)	temp (K)	1/T	total gas	N2	CO	NO	CO2	N2O
120	393	2.54						
100	373	2.68						
80	353	2.83	0.038	0.030	0.000	0.000	0.007	0.000
60	333	3.00	0.025	0.018	0.000	0.000	0.004	0.000

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This work was performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore National Laboratory under contract number W-7405-ENG-48.